

इंटरनेट

मानक

### Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 5915 (1970): Single Texture Rubberized Waterproof Fabrics [PCD 13: Rubber and Rubber Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



BLANK PAGE



**IS 1 5915 - 1970**

( Reaffirmed 2001 )

*Indian Standard*  
**SPECIFICATION FOR SINGLE TEXTURE  
RUBBERIZED WATERPROOF FABRICS**

( Second Reprint NOVEMBER 1985 )

UDC 677.866.064



© Copyright 1971

**INDIAN STANDARDS INSTITUTION**  
**MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG**  
**NEW DELHI 110002**

Gr 6

June 1971

# *Indian Standard*

## SPECIFICATION FOR SINGLE TEXTURE RUBBERIZED WATERPROOF FABRICS

Treated Fabrics Sectional Committee, CETDC 3

<i>Chairmen</i>	<i>Representing</i>
SHRI RAMAN M. PATEL	The Bhor Industries Pvt Ltd, Bhor
<i>Members</i>	
SHRI R. V. BEHERAY SHRI A. BOSE CHEMIST AND METALLURGIST REGIONAL OFFICER (CM-1), RESEARCH, DESIGN AND STANDARDS ORGANIZATION, CHITTARANJAN ( <i>Alternate</i> )	Kilburn & Co Pvt Ltd, Calcutta Bengal Waterproof Works (1940) Ltd, Calcutta Integral Coach Factory, Madras
SHRI B. H. DALAL SHRI M. KUMARAN ( <i>Alternate</i> )	Ministry of Defence (DGI)
SHRI S. L. GANDHI SHRI B. S. MATHUR ( <i>Alternate</i> )	Ministry of Defence (R & D)
CAPT R. D. KOHLI SHRI R. PARIKSHIT	The Shipping Corporation of India Ltd, Bombay Directorate General of Technical Development, New Delhi
SHRI I. L. PATEL SHRI B. P. SENGUPTA ( <i>Alternate</i> )	Directorate General of Supplies & Disposals, New Delhi
SHRI P. D. RAJE SHRI P. RAMDAS SHRI K. S. SADAGOPAN SHRI J. K. AGARWAL ( <i>Alternate</i> )	The Association of State Road Transport Under- takings, New Delhi Central Warehousing Corporation, New Delhi Dharampur Leathercloth Co Ltd, Dharampur
SHRI N. B. SENGUPTA SHRI S. P. MULLICK ( <i>Alternate</i> )	National Test House, Calcutta
SHRI A. C. SRIVASTAVA SHRI D. DAS GUPTA, Director (Chem)	Directorate General of Mines Safety, Dhanbad Director General, ISI ( <i>Ex-officio Member</i> )

*Secretary*

DR A. K. BHATTACHARYA  
Deputy Director (Chem), ISI

(Continued on page 2)

**INDIAN STANDARDS INSTITUTION**  
**MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG**  
**NEW DELHI 110002**

**IS : 5915 - 1970**

( *Continued from page 1* )

**Rubberized Fabrics Subcommittee, CETDC 3 : 5**

<i>Convener</i>	<i>Representing</i>
<b>SHRI A. BOSE</b>	<b>Bengal Waterproof Works ( 1940 ) Ltd, Calcutta</b>
<i>Members</i>	
<b>SHRI S. K. CHAKRAVARTY</b>	<b>Hindustan Rubber Works Ltd, Calcutta</b>
<b>SHRI A. K. SEN ( <i>Alternate</i> )</b>	
<b>SHRI B. H. DALAL</b>	<b>Ministry of Defence ( DGI )</b>
<b>SHRI R. K. GUPTA ( <i>Alternate</i> )</b>	
<b>SHRI A. GHOSH</b>	<b>National Test House, Calcutta</b>
<b>SHRI V. D. FENDESE</b>	<b>Swastik Rubber Products Ltd, Poona</b>
<b>SHRI B. ROY</b>	<b>East India Rubber Works Pvt Ltd, Calcutta</b>
<b>SHRI B. P. SEN GUPTA</b>	<b>Directorate General of Supplies &amp; Disposals, New Delhi</b>
<b>SHRI G. M. GADRE ( <i>Alternate</i> )</b>	

*Indian Standard*  
**SPECIFICATION FOR SINGLE TEXTURE  
RUBBERIZED WATERPROOF FABRICS**

**0. FOREWORD**

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 16 May 1970, after the draft finalized by the Treated Fabrics Sectional Committee had been approved by the Chemical Division Council, Mechanical Engineering Division Council and Textile Division Council.

**0.2** Single texture rubberized fabrics coated on one or both sides are used for making a variety of articles, such as rain coats, waterproof shoes, gloves, caps and waterproof covering for infant and sick beds. Acid and alkali resistant rubberized fabrics are used for making laboratory aprons. Fabrics where two textile plies are bonded by a layer of rubber in between are being covered by another standard because of some inherent difference in characteristics and requirements.

**0.3** This standard contains clauses **4.1, 4.2.2, 4.3.2, 4.3.3** and **4.3.9** which call for agreement between the purchaser and the supplier.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

**1. SCOPE**

**1.1** This standard prescribes the requirements and methods of test for single texture rubberized waterproof fabrics for general purposes, and for making acid and alkali resistant aprons.

**2. TERMINOLOGY**

**2.1** For the purpose of this standard, the definitions of terms given in IS : 2244-1965† shall apply.

\*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to treated fabrics.

### **3. TYPES, QUALITIES AND GRADES**

**3.1 Types** — Rubberized waterproof fabrics shall be of two types.

**3.1.1** Type 1 shall be single-faced material where the proofing shall be applied only on one side of the base fabric.

**3.1.2** Type 2 shall be double-faced material where the proofing shall be applied on both sides of the base fabric.

**3.2 Qualities** — Each of the above types of rubberized waterproof fabrics shall be made available in two qualities based on the minimum rubber polymer content of the proofing by weight as indicated in **3.2.1** and **3.2.2**.

**3.2.1 Quality 1** — Minimum rubber polymer content of the proofing shall be 55 percent by weight, when tested according to Appendix A.

**3.2.2 Quality 2** — Minimum rubber polymer content of the proofing shall be 35 percent by weight, when tested according to Appendix A.

**3.3 Grades** — Rubberized fabrics of each quality shall be made available in two grades as described in Table 1.

### **4. REQUIREMENTS**

**4.1 Base Fabric** — Base fabric for the manufacture of rubberized fabric shall be made of cotton or viscose staple or other suitable textile material as agreed to between the purchaser and the supplier. In the case of coloured single-faced rubberized fabrics, the base fabric shall be dyed to the required shade if specified by the purchaser.

#### **4.2 Proofing**

**4.2.1** The proofing (coating) shall be made from natural rubber or suitable vulcanizable synthetic rubber or a combination thereof, compounded with the necessary ingredients.

**4.2.2** It shall not contain substances such as copper and manganese compounds in such amounts as to have a deleterious action on rubber. Reclaimed rubber, vulcanized waste and rubber substitutes shall not be used if so required by the purchaser.

**4.2.3** The proofing shall be non-irritant to the skin and shall not contain any ingredients which may be harmful to persons with whom it may come into contact.

#### **4.3 Finished Fabric**

**4.3.1 General** — The finished material shall be soft, pliable, smooth and free from cuts, embedded foreign matter, surface irregularities and other mechanical defects. The double-faced rubberized fabrics shall have approximately equal amounts of proofing on both sides.



**4.3.2 Length** — The length of each piece of rubberized fabric shall not be less than 30 metres unless otherwise agreed to between the purchaser and the supplier. There shall be no joints in any single piece. The length shall be determined in accordance with IS : 1954-1961\*.

**4.3.3 Width** — The usable width of the rubberized fabric shall be as agreed to between the purchaser and the supplier. The unproofed portion, if any, near either selvedge shall not be more than 10 mm in width. The width shall be determined in accordance with IS : 1954-1961\*.

**4.3.4 Waterproofness** — The finished material shall be such that, when tested by the method prescribed in Appendix E of IS : 1389-1959†, the surface in contact with the air shall show no water globules or wet patches, when a constant head of water, as indicated below, is maintained for 30 minutes:

Type/Quality/Grade of Fabric	Head of Water, cm
Qualities 1 and 2 and Grades A and B of Type 1	30
Quality 1, Grade A of Type 2	
Quality 1, Grade B of Type 2	60
Quality 2, Grade A of Type 2	
Quality 2, Grade B of Type 2	90

**4.3.5 Accelerated Ageing Test** — When the finished material is tested at a temperature of  $90 \pm 1^\circ\text{C}$  for 168 hours in accordance with the method described in 4.3 of IS : 3400 (Part IV)-1965‡, the rubber proofing shall not become stiff, softened or tacky, nor shall it show appreciable discoloration or be easily detachable from the base fabric. When the aged sample is tested for waterproofness as described in 4.3.4 there shall be no leakage of water through any part of it and the surface in contact with the air shall show no water globules or wet patches.

**4.3.6 Colour** — The finished material shall be white or of a suitable colour or different colours on the two sides in case of double-faced fabric as agreed to between the purchaser and the supplier. Sulphur dyes shall not be used unless otherwise agreed to between the purchaser and the supplier. The colour fastness in respect of the coloured material shall comply with the following requirements.

**4.3.6.1 Fastness to light** — The material, when tested for colour fastness as prescribed in IS : 2454-1967§, shall show a fastness rating of not less than No. 3 prescribed therein. In case of materials coloured on both sides, or in dual shade, the test shall be conducted with respect to each side of

\*Method for determination of dimensions of fabrics.

†Method for testing cotton fabrics for resistance to attack by micro-organisms.

‡Methods of test for vulcanized rubber: Part IV Accelerated ageing.

§Method for determination of colour fastness of textile materials to artificial light (xenon lamp).

## **IS : 5915 - 1970**

the material separately and the colour fastness in each case assessed for compliance with the aforesaid requirement. The test shall be applicable to coloured as well as white rubberized fabrics.

**4.3.6.2 Fastness to washing of coloured fabric**— When the rubberized fabric is tested for colour fastness to washing in accordance with IS : 3361-1965\*, the fastness rating, in respect of change in shade of the material and the degree of staining on the attached undyed pieces of cotton and woollen clothes, evaluated according to IS : 768-1956† and IS : 769-1956‡ respectively shall be not less than 5 in case of double-faced rubberized fabrics and single-faced coloured rubberized fabrics where the base fabric is not dyed. In the case of coloured single-faced rubberized fabrics where the base fabric is of dyed material, the fastness rating of the shades on the proofed and unproofed side shall be as agreed to between the purchaser and the supplier.

**4.3.7 Reaction of Aqueous Extract**— When the finished fabric is tested in accordance with the 'hot method' of IS : 1390-1961§, its aqueous extract shall neither be acidic to methyl orange nor alkaline to phenolphthalein indicator.

**4.3.8 Autoclaving Test**— A sample of the finished material tested as described in Appendix B shall not be stiffer or softer than the control piece of the material, shall not be tacky and shall show no other apparent deterioration likely to impair its usefulness.

**4.3.9 Resistance to Cold**— The proofing on the cloth shall withstand a bending test around a steel pin of 6 mm diameter without cracking, after an exposure of 5 hours to a temperature of minus  $35 \pm 1^\circ\text{C}$  or as agreed to between the purchaser and the supplier. The purchaser shall specify his option for this test.

**4.3.10 Resistance to Acid and Alkali**— When a piece,  $2.5 \times 2.5$  cm cut from any portion of the finished material, is dipped in 20 percent hydrochloric acid or 20 percent sodium hydroxide solution for 30 minutes, there shall be no sign of corrosion or damage of the proofing. The purchaser shall specify his option for the test.

**4.3.11** The finished fabric shall also comply with the requirements prescribed in Table 1.

## **5. PACKING AND MARKING**

**5.1** The material shall be securely packed in rolls so as to ensure safe transportation.

\*Method for determination of colour fastness of textile materials to washing: Test 2.

†Method for evaluating change in colour.

‡Method for evaluating staining.

§Methods for determination of pH value of aqueous extracts of textile materials.

TABLE 1 REQUIREMENTS FOR FINISHED FABRIC

(Class 4.3.11)

Sl. No.	CHARACTERISTIC	Type 1				Type 2				METHOD OF TEST, REF TO Appen- IS: chx
		Quality 1		Quality 2		Quality 1		Quality 2		
		Grade A	Grade B	Grade A	Grade B	Grade A	Grade B	Grade A	Grade B	
i)	Weight in $g/m^2$ , Max	250	375	300	400	350	400	400	800	— 1964-1961*
ii)	Proofing content in $g/m^2$ , Min	140	175	200	250	250	275	300	500	C —
iii)	Breaking strength in $kg/cm$ width, Min:									
	a) Warp	4	8	3.5	4	4	8	3.5	7	— 1969-1968†
	b) Weft	3	5	2.5	3	3	5	2.5	4	

\*Method for determination of weight per square metre and weight per linear metre of fabrics.

†Method for determination of breaking load and elongation at break of woven textile fabrics (first revision).

## **IS : 5915 - 1970**

**5.2** Each piece of the rubberized fabric shall be indelibly and legibly marked at both ends with the manufacturer's name; initials or recognized trade-mark, if any; month and year of manufacture and length of the piece in metres.

**5.3** Other identification letters shall also be stamped on the material if required by the purchaser.

**5.4** The material may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## **6. SAMPLING**

**6.1** The method of sampling of the material and the criteria for conformity shall be as prescribed in Appendix D.

## **7. TESTS**

**7.1 Test Specimens** — The test specimens shall be cut from the sample in the required number and in the appropriate manner as specified in the individual methods of tests.

**7.1.1** In cases where two or more samples are to be tested, the number of test specimens may be reduced at the discretion of the purchaser.

## **A P P E N D I X A**

*( Clauses 3.2.1 and 3.2.2 )*

### **DETERMINATION OF RUBBER POLYMER CONTENT ( INDIRECT METHOD )**

#### **A-0. GENERAL**

**A-0.1** The indirect method determines the non-rubber constituents of a product and the remainder is expressed as rubber polymer content. The non-rubber constituents are determined in accordance with **A-1**

through A-10 and rubber polymer content is calculated as given in A-10.1. The method is applicable to NR, IR, SBR and BR products and also to IIR products if they are extracted with methyl ethyl ketone rather than with acetone.

**NOTE** — The abbreviations NR, IR, SBR, IIR, BR, CR, ABR and NBR used above and hereafter denote isoprene (natural), isoprene (synthetic), styrenebutadiene, isobutylene-isoprene, butadiene, chloroprene, acrylate-butadiene and nitrile-butadiene rubbers.

## A-1. TOTAL EXTRACT

### A-1.1 Apparatus

A-1.1.1 *Extraction Apparatus* — see Fig. 1.

### A-1.2 Reagents

A-1.2.1 *Acetone* — distilled over potassium carbonate boiling between 56 to 57°C.

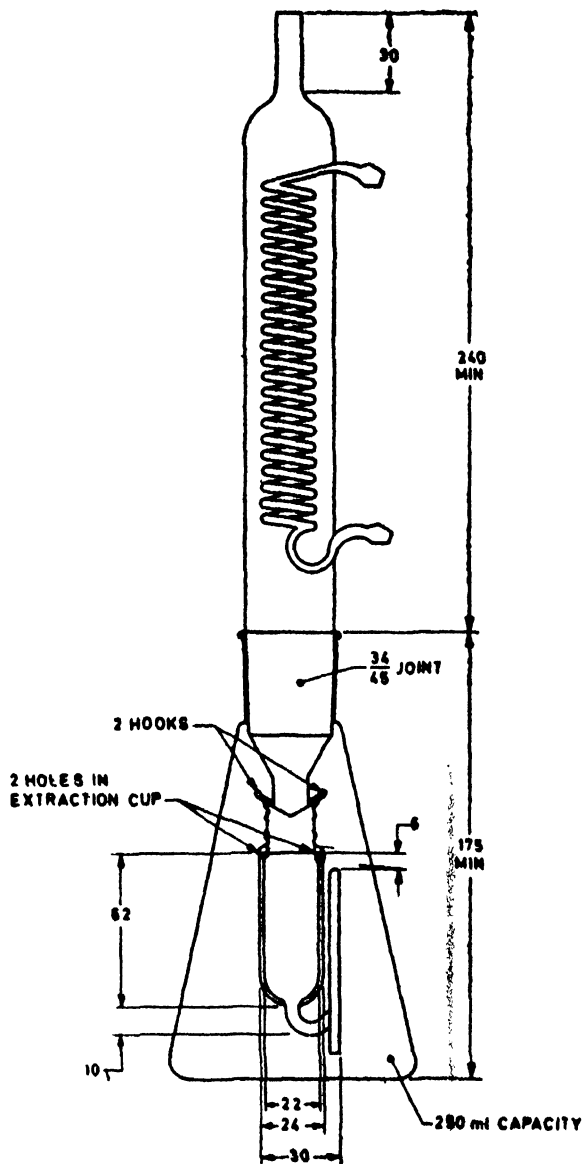
A-1.2.2 *Chloroform*

### A-1.3 Procedure

A-1.3.1 Place a weighed specimen of approximately 2 g in a filter paper. If the specimen is in the form of a sheet, cut it with scissors into strips 3 to 5 mm in width. If the specimen becomes tacky during the extraction, take care that adjacent portions are separated by paper. Fold the paper so that it will fit in the extraction cup, and suspend the cup in a weighed extraction flask containing 50 to 75 ml of a mixture consisting of 32 parts of acetone and 68 parts of chloroform by volume. ( Prior to the weighing of the extraction flask, it shall have been dried for 2 h at  $70 \pm 5^\circ\text{C}$  and cooled in a desiccator to the temperature of the balance. )

A-1.3.2 Extract the specimen continuously for 16 h, heating at a rate such that the time required to fill and empty the siphon cup will be between 2.5 and 3.5 min. ( Rubber products having a ratio of total sulphur to rubber polymer in excess of 10 percent shall be extracted for 72 h. ) Carefully note all characteristics of the extract, both when hot and cold. If the colour is black, make a chloroform extraction separately for 4 hours. dry the extract to constant weight and add the value to the result obtained for total extract.

A-1.3.3 Evaporate off the solvent over a steam bath, using a gentle current of filtered air to prevent boiling. Remove the flask from the steam bath just prior to the disappearance of the last traces of solvent to prevent loss of extract. Continue the passage of air through the flask for 10 min to remove the remaining solvent and dry the flask for 2 h in a  $70 \pm 5^\circ\text{C}$  air bath.



All dimensions in millimetres.

FIG. 1 EXTRACTION APPARATUS WITH GLASS CONDENSER AND GROUND JOINT

**A-1.3.4** Cool in a desiccator to the temperature of the balance and weigh. Save the extracted rubber for further tests that require the use of an extracted specimen.

**A-1.4 Calculation**

$$\text{Total extract percent} = \frac{A}{B} \times 100$$

where

*A* = weight in g of extract,

*B* = weight in g of specimen used.

**A-2. ALCOHOLIC POTASH EXTRACT**

**A-2.1 Reagent**

**A-2.1.1 Alcoholic Potash Solution** — Prepare a 1 N alcoholic potassium hydroxide solution by dissolving the required amount of potassium hydroxide in absolute ethanol that has been purified as follows:

Dissolve 1.5 g of silver nitrate in 3 ml of water and add it to one litre of alcohol. Dissolve 3 g of potassium hydroxide in the smallest amount of hot water, cool, add it to the silver nitrate solution, and shake thoroughly. Allow the solution to stand for at least 24 h, filter, and distil.

**Note** — Absolute ethanol denatured with 10 percent by volume of methanol may also be used.

**A-2.1.2 Congo Red Paper**

**A-2.1.3 Ethanol** — 95 percent.

**A-2.1.4 Ether**

**A-2.2 Procedure**

**A-2.2.1** Remove the specimen remaining after the determination of or total extract from its wrapping material while wet with solvent and dry the rubber at  $70 \pm 5^\circ\text{C}$  to remove the solvent.

**A-2.2.2** Transfer to a 200-ml Erlenmeyer flask, add 50 ml of alcoholic potash solution, and heat under a reflux condenser for 4 h. In the case of hard rubber, continue the heating for 16 h or more.

**A-2.2.3** Filter into a 250-ml beaker, wash with two 25-ml portions of boiling alcohol and then with three 25-ml portions of boiling water, and evaporate the filtrate just to dryness. Use about 75 ml of water to transfer the dried filtrate residue to a separatory funnel. Acidify the solution with hydrochloric acid (1 : 3), testing with Congo red paper.

**A-2.2.4** Extract with four 25-ml portions of ether, unless the fourth portion should be colored, when the extraction shall be continued until no further quantity can be removed. Unite the ether fractions and wash thoroughly with water until free of acid (two washings are generally sufficient).

**A-2.2.5** Filter the ether solution through a plug of previously washed absorbent cotton into a weighed flask and wash the separatory funnel and the cotton plug with ether. Evaporate the ether on a steam bath, using a gentle current of filtered air to prevent boiling. Remove the flask from the steam bath just prior to the disappearance of the last traces of solvent and continue the passage of air for 10 minutes. Dry the flask at  $100 \pm 5^{\circ}\text{C}$  to constant weight, cool and weigh.

### **A-2.3 Calculation**

$$\text{Alcoholic potash extract, percent} = \frac{A}{B} \times 100$$

where

*A* = weight in g of extract, and

*B* = weight in g of specimen used.

## **A-3. TOTAL SULPHUR ( ZINC-NITRIC ACID METHOD )**

**A-3.0 General** — This method covers the determination of all the sulphur, except that contained in barium sulphate, in a sample of rubber product or in the fillers obtained from a rubber product. If acid-soluble barium salts, antimony sulphide, or lead compounds are present, this method will give erroneous results, in which case the fusion method ( *see A-4* ) should be used. This method may be used for the determination of organic plus inorganic sulphur on an extracted sample, total plus inorganic sulphur on an unextracted sample, or inorganic sulphur in fillers. If it is used for determination of total or organic sulphur plus inorganic sulphur, it shall also be used for determination of inorganic sulphur ( *see A-5* ). The method is applicable to NR, SBR, BR, IR and CR products and to total plus inorganic sulphur determination of NBR products.

### **A-3.1 Reagents**

**A-3.1.1 Barium Chloride Solution** ( 100 g  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ /litre ) — Dissolve 100 g of barium chloride (  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  ) in water, add two to three drops of hydrochloric acid, and dilute to 1 litre. If there is any insoluble matter or cloudiness, heat the solution overnight on a steam bath and filter.

**A-3.1.2 Bromine, Saturated Water Solution**

**A-3.1.3 Nitric Acid, Fuming**



**A-3.1.4 Picric Acid, Saturated Solution****A-3.1.5 Potassium Chlorate (  $KClO_3$  )**

**A-3.1.6 Zinc-Nitric Acid Solution** — Add 200 g of zinc oxide (  $ZnO$  ) to one litre of nitric acid slowly and with caution against spattering.

**A-3.2 Procedure**

**A-3.2.1** Place 0.5 g of soft rubber or 0.2 g of hard rubber ( extract the specimen with acetone or acetone-chloroform mixture if organic sulphur is to be determined ) in a 500-ml Erlenmeyer destruction flask of chemically resistant material. Add 10 ml of zinc-nitric acid solution and moisten the sample thoroughly. Let stand at least for 1 h; overnight if convenient. By so doing the sample becomes partly decomposed; this permits the addition of fuming nitric acid with no danger of ignition of the sample. Add 15 ml of fuming nitric acid and whirl the flask rapidly to keep the sample immersed to avoid ignition. With some samples it may be necessary to cool the flask under running water.

**A-3.2.2** When the dissolution of the rubber appears to be complete, add 5 ml of a saturated water solution of bromine and slowly evaporate the mixture to a foamy syrup. ( For the determination of sulphur in unvulcanized mixtures, use 3 ml of bromine in place of bromine water. )

**A-3.2.3** If organic matter or carbon remains at this point, add a few millilitres of fuming nitric acid and a few crystals of potassium chlorate ( *caution* ) and evaporate at a boil. Repeat this operation until all carbon is removed and the solution is clear, colourless, or light yellow.

**A-3.2.4** At this point either of the following procedures may be used:

*Procedure A* — Place the flask on an asbestos gauze and evaporate the mixture to dryness over a burner. Then bake the mixture at the highest temperature of the burner until all nitrates are decomposed and no more nitrogen oxide fumes can be detected. The flask and its contents shall be carefully annealed after this procedure by gradually decreasing the flame or by placing the flask on successively cooler sources of heat.

*Procedure B* — Evaporate the mixture, cool, add 10 ml of hydrochloric acid, and evaporate to dryness, avoiding spattering. Repeat this procedure once, or more than once if oxides of nitrogen are still evolved.

**A-3.2.5** Cool the flask, add 50 ml of hydrochloric acid ( 1 : 6 ) and digest hot until dissolution is as complete as possible. Filter while hot. Wash the filter and dilute the filtrate and washings to about 300 ml. Add 10 ml of saturated picric acid solution, heat to  $90^{\circ}C$ , and precipitate the sulphate by dropwise addition of barium chloride solution while stirring vigorously. Digest the precipitate overnight, preferably at  $60$  to  $80^{\circ}C$ ,

using a watch glass to cover the beaker. Filter the barium sulphate and wash with water until the filter is colourless. Dry, ash, and finally ignite the precipitate at 650 to 900°C, with free access to air, to constant weight. Cool in a desiccator and weigh.

#### A-3.2.6 Calculation

$$\text{Sulphur, percent} = \frac{A \times 0.1373}{B} \times 100$$

where

*A* = weight in g of barium sulphate, and

*B* = weight in g of specimen used.

### A-4. TOTAL SULPHUR (FUSION METHOD)

**A-4.1 General** — This method covers the determination of organic, total or inorganic sulphur in rubber compounds when acid-soluble barium salts, antimony sulphide, or lead compounds are present. All of the sulphur in the sample, including that present in barium sulphate is determined when this method is used. When this method is used for determination of organic or total sulphur it shall also be used for the determination of inorganic sulphur (see A-5). Total plus inorganic sulphur shall be determined on an unextracted specimen; organic plus inorganic sulphur on an extracted specimen. The method is applicable to NR, SBR, BR, IR and CR products and to the determination of total plus inorganic sulphur on NBR products.

#### A-4.2 Reagents

**A-4.2.1 Nitric Acid-Bromine Solution** — Add a considerable excess of bromine to nitric acid so that a layer of bromine is present in the reagent bottle. Shake thoroughly and allow to stand 24 h before using.

**A-4.2.2 Sodium Carbonate** ( $\text{Na}_2\text{CO}_3$ )

**A-4.2.3 Sodium Carbonate Solution** — 50 g/litre.

#### A-4.3 Procedure

**A-4.3.1** Place 0.5 g of rubber in a porcelain crucible of about 75-ml capacity. The specimen shall have been extracted with acetone or acetone-chloroform mixture (see A-1) if organic plus inorganic sulphur is to be determined. Add 15 ml of the nitric acid-bromine mixture, cover the crucible with a watch glass, and let it stand for 1 h in the cold. Heat for 1 h on the steam bath, remove the cover, rinse it with a little water, and evaporate to dryness.

**A-4.3.2** Add 3 ml of nitric acid, cover, warm for a short time on the steam bath and then allow to cool. Carefully add in small portions, by means of a

glass spatula, 5 g of sodium carbonate. Raise the watch glass only high enough to permit the introduction of the spatula. Allow the sodium carbonate to slide down the side of the crucible, as it shall not be dropped directly into the acid. Rinse the watch glass with 2 or 3 ml of hot water and stir the mixture thoroughly with a glass rod. Digest for a few minutes, spread the mixture halfway up the side of the crucible to facilitate drying, and dry on a steam bath. Fuse the mixture by heating over a sulphur-free flame.

**A-4.3.3** Place the crucible in an inclined position on a wire triangle and start the ignition over a low flame. The tendency for the organic matter to burn too briskly may be controlled by judicious use of the stirring rod with which the burning portion is scraped away from the rest. When part of the mass is burned white, work a fresh portion into it until all of the organic matter is destroyed. It is necessary to hold the edge of the crucible with tongs. Towards the last half of the operation the flame should be increased. It is unnecessary to heat the crucible to redness. With care, a crucible can be used for at least 10 to 12 fusions.

**A-4.3.4** After a fusion, allow the crucible to cool. Place it in a 400-ml beaker, add sufficient water to cover the crucible (about 125 ml), and digest on the steam bath or plate for at least 2 h.

**A-4.3.5** Filter the solution into a covered 400-ml beaker containing 5 ml of hydrochloric acid and wash the residue thoroughly with hot sodium carbonate solution. A qualitative test for barium may be made on the residue, but no analysis for barium or correction because of its presence is necessary, unless a detailed ash analysis is desired. Acidify the filtrate to indicator paper with hydrochloric acid and add 2 ml in excess. Precipitate barium sulphate and complete the determination as described in A-3.

#### **A-4.3.6** Calculation

$$\text{Sulphur, percent by weight} = \frac{A \times 0.1373}{B} \times 100$$

where

$A$  = weight in g of barium sulphate, and

$B$  = weight in g of specimen used.

## **A-5. INORGANIC SULPHUR**

**A-5.1 General Type** — This method covers the determination of inorganic sulphur in rubber products when no antimony is present. The sample is ashed and sulphur determined in the ash by the fusion method (see A-4) if acid-soluble barium salts or lead are present, or by the zinc-nitric acid method in the absence of acid-soluble barium salts. The same method shall be used for the inorganic sulphur determination as is

## IS : 5915 - 1970

used for the determination of organic sulphur or total sulphur. The method is applicable to NR, SBR, BR, IR, CR, NBR and IIR products.

### A-5.2 Reagents

#### A-5.2.1 Nitric Acid-Bromine Solution.

**A-5.3 Procedure** — Extract a 1.0-g specimen with acetone or with acetone-chloroform mixture (see A-1). Dry the sample, place in a porcelain crucible of about 75 ml capacity and distil off the rubber in a muffle furnace, using a maximum temperature of 450°C. A burner may be used for ashing if the sample is not allowed to catch fire. A wire gauze under the crucible will aid in preventing combustion. The carbon need not be completely burned off in this ignition. If acid soluble barium salts and lead are absent, add 3 ml of nitric acid bromine mixture to the ash, cover with a watch glass, and heat for 1 h. Transfer the contents of the crucible, with washing, into a 500-ml Erlenmeyer destruction flask of chemically resistant material and evaporate to dryness. Proceed with the determination of sulphur as described in A-3.2.3 to A-3.2.5. In the presence of acid-soluble barium salts and lead, determine the sulphur by treating the ash in accordance with A-4.

### A-5.4 Calculation

$$\text{Inorganic sulphur, percent} = \frac{A \times 0.1373}{B} \times 100$$

where

$A$  = weight in g of barium sulphate, and

$B$  = weight in g of specimen used.

## A-6. ORGANIC SULPHUR

**A-6.1** Organic sulphur is calculated by subtracting inorganic sulphur as obtained in A-5 from total sulphur as obtained in A-3 or A-4 as applicable.

## A-7. FILLERS ( REFEREE ASH METHOD )

**A-7.1 General** — This method is intended for settling disagreements on fillers content by the ashing method. It is also useful for ashing rubber products for determination of inorganic sulphur in the absence of antimony or for ash analysis.

**A-7.1.1** The method is not accurate for rubbers containing halogens when zinc compounds or other metal compounds that form volatile halides are present and shall not be used if an analysis of these metals in the ash is required.

**A-7.1.2** The method may be used for preparing samples for ash analysis on rubber products not containing halogens or antimony sulphide. However, if the sample contains carbonates that decompose at 550°C or clays or silicates that will lose water at this temperature, the ash content value will not be highly precise and will not represent the original amount of inorganic fillers present in the sample.

## **A-7.2 Apparatus**

**A-7.2.1 Crucibles** — porcelain or silica, approximately 50-ml capacity.

**A-7.2.2 Electric Muffle Furnace** — with controls necessary to hold the temperature at 550 ± 25°C.

**A-7.2.3 Calibrated Thermocouple and Temperature Recording Device**

## **A-7.3 Procedure**

**A-7.3.1** Weigh a 1-g specimen of the sample into an ignited, weighed crucible.

**A-7.3.2** Adjust the temperature of the muffle furnace to 550 ± 25°C, place the crucible in the furnace, and close the door completely. When more than one crucible is to be placed in the furnace, the crucible shall be placed on a tray and put into the furnace. The door shall be closed immediately and not opened for 1 h. After 1 h, open the furnace door 3 to 5 cm and continue heating for 30 minutes or until all carbonaceous material is burned off.

**Note** — If a referee ash determination is required, the temperature shall be determined by placing the calibrated thermocouple sensing element at approximately the geometric centre of the furnace cavity. The temperature shall be adjusted to be within the specified range. No more than two crucibles shall be placed in the oven and they shall be positioned directly below the thermocouple.

**A-7.3.3** Remove the crucible from the furnace, cool in a desiccator, and weigh.

## **A-7.4 Calculation**

$$\text{Ash, percent} = \frac{A - B}{C} \times 100$$

where

*A* = weight in g of ash and crucible,

*B* = weight in g of crucible, and

*C* = weight in g of specimen.

IS : 5915 - 1970

## A-8. CARBON BLACK

### A-8.1 General

**A-8.1.1** This method covers the determination of carbon black by a nitric acid digestion method. It is applicable only to the 'R' family of rubbers containing an unsaturated carbon chain.

**A-8.1.2** Application to any other rubber type may be possible if the rubber is degraded to fragments soluble in water, acetone, or chloroform. The method shall be used on such other rubbers only if adequate testing of known compounds has demonstrated the usefulness of the method.

### A-8.2 Reagents

**A-8.2.1** *Acetone*

**A-8.2.2** *Chloroform*

**A-8.2.3** *Sodium Chromate Solution* — 100 g  $\text{Na}_2\text{CrO}_4$ /litre.

**A-8.2.4** *Sodium Hydroxide Solution* — 175 g NaOH/litre.

**A-8.2.5** *Sodium Hydroxide Solution* — 300 g NaOH/litre.

### A-8.3 Procedure

**A-8.3.1** Extract a 0.5-g specimen with acetone-chloroform mixture in accordance with A-1.

**A-8.3.2** Transfer the specimen to a 250-ml beaker and heat on the steam bath until it no longer smells of chloroform. Add a few millilitres of nitric acid and allow to stand for about 10 min. Add 50 ml more of nitric acid, taking care to wash down the sides of the beaker, and heat on the steam bath for at least 1 h. At the end of this time there should be no more bubbles or foam on the surface. Pour the liquid, while hot, into a Gooch crucible, taking care to keep as much as possible of the insoluble material in the beaker. Filter slowly with gentle suction and wash well by decantation with hot nitric acid. (Caution — Empty the filter flask) (see Note). Wash with acetone and a mixture of equal parts of acetone and chloroform until the filtrate is colourless. Digest the insoluble material, which has been carefully retained in the beaker, for 10 min on the steam bath with 35 ml of sodium hydroxide solution (300 g/litre). This treatment with alkali may be omitted if silicates are absent. Dilute to 60 ml with hot water and heat on the steam bath. Filter the solution of alkali and wash well with hot sodium hydroxide solution (175 g/litre).

**NOTE** — The filtration may be materially aided, particularly with some synthetic rubber products, by partial or complete neutralization of the nitric acid solution with sodium hydroxide. Partial neutralization together with the addition of trivalent cations or anions may also aid agglomeration of the carbon black particles if they are too well dispersed to filter.

**A-8.3.3** Next, wash the residue about four times with hot hydrochloric acid. Neutralize the last washing with sodium hydroxide and test for the presence of lead with sodium chromate solution. If lead is present, continue to wash with hot hydrochloric acid and, finally, wash with warm hydrochloric acid (1:7). Remove the crucible from the funnel, taking care that the outside is perfectly clean, dry it in an air bath for 1½ h at 110°C, cool, and weigh; call this weight *a*. Burn off the carbon at a dull red heat (550 to 600°C) and reweigh; call this weight *b*. The difference in weight represents approximately 105 percent of the carbon originally present in the form of carbon black.

#### **A-8.4 Calculation**

$$\text{Carbon black, percent} = \frac{a - b}{1.05 \times c} \times 100$$

where

*c* = weight in g of specimen used.

### **A-9. NITROGEN (CALCULATED AS GLUE)**

**A-9.1 General** — This method is intended for use in the determination of glue when it is used as a filler in rubber products other than NBR products. The method may also be used to determine nitrogen from NBR polymer present in a product. The calculations in this section are designed for determination of glue in NR products. In the absence of other nitrogenous material, the method may be applied to synthetic rubber products without correction for the nitrogen content of the rubber polymer. A slight error will result from nitrogen content of antioxidants and accelerators present after extraction. When glue is found to be present in a NR product and determined by this method by calculation from nitrogen content, a correction shall be made for the natural protein in NR as shown in the calculations. The correction shall be made by the method of approximation. The rubber as compounded is calculated on the basis of the total nitrogen as glue. The glue content is then corrected on the assumption that the rubber as compounded contains 0.4 percent protein nitrogen and this value is used to calculate a more exact figure for rubber hydrocarbon.

#### **A-9.2 Reagent**

**A-9.2.1** *Copper Sulphate* (  $\text{CuSO}_4$  )

**A-9.2.2** *Paraffin*

**A-9.2.3** *Sodium Hydroxide Solution* — ( 750 g NaOH/litre ).

**A-9.2.4** *Sodium Hydroxide Standard Solution* — Prepare and standardize a 0.1 N sodium hydroxide ( NaOH ) solution.

IS : 5915 - 1970

**A-9.2.5 Sodium Sulphate** ( $\text{Na}_2\text{SO}_4$ )

**A-9.2.6 Sulphuric Acid, Standard Solution** — Prepare and standardize a 0.1 N sulphuric acid ( $\text{H}_2\text{SO}_4$ ) solution.

**A-9.2.7 Zinc** — granulated.

**A-9.3 Procedure** — Extract a 2-g specimen with acetone for 8 hours. Remove the solvent from the specimen, and transfer the latter from the filter paper to a 750-ml Kjeldahl flask. Add 25 to 30 ml of sulphuric acid, 10 to 12 g of sodium hydroxide and about 1 g of copper sulphate. Heat gently until the first vigorous frothing ceases; then raise the heat gradually until the liquid boils. Continue the boiling until the solution becomes clear. Allow the flask to cool, dilute carefully with 150 ml of water, and again allow to cool. Add 10 ml of sodium hydroxide solution (750 g/litre), pouring it carefully down the side of the flask so that it does not mix immediately with the acid solution. Add about 1 g of granulated zinc to prevent bumping and a piece of paraffin the size of a pea to diminish frothing. Connect the flask quickly with a condenser, the delivery tube of which dips into a 500-ml Erlenmeyer flask, containing 50 ml of 0.1 N sulphuric acid diluted to about 100 ml. Carefully swirl the flask to mix the contents and start to heat gently, increase the flame as the danger of foaming over diminishes, and finally boil briskly until about one-half of the liquid has passed over into the receiver. Add methyl red solution and titrate the excess acid by means of 0.1 N sodium hydroxide solution.

#### A-9.4 Calculation

$$\text{Total nitrogen as gluc, percent} = \frac{(AN - BN') \times 0.014 \times 6.5}{C} \times 100$$

where

$A$  = volume in ml of sulphuric acid used,

$N$  = normality of sulphuric acid,

$B$  = volume in ml of sodium hydroxide used,

$N'$  = normality of sodium hydroxide, and

$C$  = weight in g of specimen used.

### A-10. RUBBER POLYMER CONTENT

#### A-10.1 Calculation

$$\text{Rubber polymer content, percent} = A (100 - B)$$



where

*A* = factor as listed below:

<i>Rubber</i>	<i>Factor A</i>
NR	94/97
IR	1.00
SBR*	1.00
BR	1.00
IIR	1.00

*B* = sum of percentage, of total extract, alcoholic potash extract, organic sulphur, ash, carbon black and glue determined in accordance with **A-1**, **A-2**, **A-6**, **A-7**, **A-8** and **A-9** respectively.

## APPENDIX B

( Clause 4.3.8 )

### METHOD FOR AUTOCLAVING TEST

#### B-1. TEST SPECIMENS

**B-1.1** Each test piece shall measure not less than 10 × 5 cm.

#### B-2. APPARATUS

**B-2.1** The apparatus shall consist of an autoclave containing steam at 1.1 kg per square cm gauge pressure. The space in the autoclave shall not contain more than one part of air to 1 000 parts of steam.

#### B-3. PROCEDURE

**B-3.1** Keep the test pieces inside the autoclave for 20 minutes, then remove the pieces from the autoclave and allow them to cool to room temperature. Repeat the above autoclaving process after a period of two hours, then remove the test pieces from the autoclave and allow them to cool to room temperature and dry in the air. Examine the test pieces for any sign of softening, stiffening, tackiness, brittleness or loss of other rubber like properties of the coating.

\*Containing 23.5 percent bound styrene and not oil extended.

## APPENDIX C

( Table 1 )

### DETERMINATION OF CONTENT PROOFING

#### C-1. REAGENT

**C-1.1 Solvent Capable of Swelling or Dissolving Cured Polymer Present in the Sample** — The choice of a suitable solvent depends upon the nature of the polymer and upon the properties of the textile base.

#### C-2. PROCEDURE

**C-2.1** Cut 4 test pieces each approximately 10 cm square equally spaced across the width of the fabric with the 2 test pieces centered 15 cm from the selvages. After conditioning at  $27 \pm 2^\circ\text{C}$  and  $65 \pm 2$  percent relative humidity immerse each test piece in the solvent and heat under the reflux until the proofing is dissolved or swollen thoroughly (generally about 1 hour) taking care not to allow the temperature to exceed  $160^\circ\text{C}$ . Remove the test piece from the solvent and carefully scrape off any swollen proofing with a spatula. Repeat the treatment using fresh solvent on each occasion until the fabric is free from rubber. Squeeze the fabric to remove solvent and rinse with light petroleum hydrocarbon solvent until free from the original solvent. Dry 1 hour at  $105\text{-}110^\circ\text{C}$  and then condition for 24 hours. Transfer to a weighing bottle and determine the weight of the fabric.

**C-2.2** Ash the fabric at a low temperature in a previously ignited and weighed silica crucible and determine the weight of the ash.

#### C-3. EXPRESSION OF RESULTS

**C-3.1** The proofing, content,  $\text{g/m}^2$  ( as determined on 10 cm square test piece )  $= 100 ( a - b + c )$

where

$a$  = weight in g of original test piece,

$b$  = weight in g of fabric after removal of proofing, and

$c$  = weight in g of ash of fabric.

## APPENDIX D

( Clause 6.1 )

## SAMPLING OF RUBBERIZED FABRICS

## D-1. SAMPLING OF RUBBERIZED FABRICS

**D-1.1 Lot**— In any consignment, all the rolls of rubberized fabrics of same type, quality and grade shall be grouped together and each such group shall constitute a lot.

**D-1.1.1** The conformity of the lot to the requirements of the specification shall be ascertained for each lot separately. The number of rolls ( $n$ ) to be selected from a lot shall depend on the size of the lot ( $N$ ) and shall be in accordance with Table 2.

---

**TABLE 2 NUMBER OF ROLLS TO BE SELECTED FOR SAMPLING**

LOT SIZE	NUMBER OF ROLLS TO BE SELECTED
$N$	$n$
(1)	(2)
Up to 50	2
51 „ 100	3
101 „ 200	4
201 „ 300	5
301 and above	7

---

**D-1.1.2** The rolls shall be selected at random and to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted subject to agreement between the purchaser and the supplier:

Arrange all the rolls in the lot in a systematic manner and starting from any roll, count them as 1, 2, 3,....., etc, up to  $r$  and so on, where  $r$  is the integral part of  $N/n$  ( $N$  being the lot size and  $n$  the sample size). Every  $r$ th roll thus shall be withdrawn till the requisite number of rolls is obtained.

**D-1.2 Number of Tests**

**D-1.2.1** From each of the rolls selected according to D-1.1.2, one metre length of rubberized fabric shall be cut, care being taken to exclude not less than 0.25 metre length of fabric from either end. The test specimens

**IS : 5915 - 1970**

necessary for the various tests specified in the standard shall be cut from the lengths of rubberized fabric thus obtained.

**D-1.2.2** Tests for the determination of all the characteristics specified in the standard, shall be conducted on the test specimens cut from these metre-lengths as obtained in **D-1.2.1**.

**D-1.3 Criteria for Conformity**

**D-1.3.1** The lot shall be declared as conforming to the specification if for each of the characteristics the test results on all the individual metre-lengths ( *see* **D-1.2.1** ) are found to be within the limits of the specification.

**D-1.3.2** If specimens taken from these lengths fail in one or more tests, each such test shall be repeated twice. For this purpose, two further metre-lengths shall be cut from the same roll as the failing metre-length and specimens shall be cut from each of them so that duplicate tests may be conducted in respect of each failure. If all the retests have passed, the lot shall be declared as conforming to the specification, otherwise not.



# INDIAN STANDARDS INSTITUTION

## Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 3310131, 331137\*

Telegrams : Manaksanstha  
(Common to all offices)

## Regional Offices :

Telephone

\*Western : Manakalaya, E9 MIDC, Marol, Andheri (East). 6 32 92 95  
BOMBAY 400093

†Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road  
Maniktola, CALCUTTA 700054 35 24 99

Southern : C. I. T. Campus, MADRAS 600113 41 24 42

Northern : B69 Phase VII, Industrial Focal Point,  
S. A. S. NAGAR 160051 (Punjab) 8 73 28

## Branch Offices :

Pushpak, Nurmohamed Shalkh Marg, Khanpur, AHMADABAD 380001 { 2 63 48  
2 63 49

'F' Block, Unity Bldg, Narasimharaja Square, BANGALORE 560002 22 48 05

Gangotri Complex, Bhadbhada Road, T. T. Nagar, BHOPAL 462003 6 27 16

22E Kalpana Area, BHUBANESHWAR 751014 5 36 27

5-8-56C L. N. Gupta Marg, HYDERABAD 500001 22 10 83

R14 Yudhister Marg, C Scheme, JAIPUR 302005 6 98 32

117/418 B Sarvodaya Nagar, KANPUR 208005 4 72 92

Patliputra Industrial Estate, PATNA 800013 6 23 05

Hantex Bldg (2nd Floor), Rly Station Road, TRIVANDRUM 695001 32 27

## Inspection Office (With Sale Point) :

Institution of Engineers (India) Building, 1332 Shilpi Nagar, 6 24 35  
PUNE 411005

\*Sales Office in Bombay is at Novelty Chambers, Grant Road, 69 65 26  
Bombay 400007

†Sales Office in Calcutta is at 5 Chowringhee Approach, P. O. Princep Street, Calcutta 700072 27 65 98

**AMENDMENT NO. 1    DECEMBER 1973**  
**TO**  
**IS : 5915-1970 SPECIFICATION FOR SINGLE**  
**TEXTURE RUBBERIZED WATERPROOF**  
**FABRICS**

**Addendum**

( Page 4, clause 4.2.3 ) — Add the following new clause after 4.2.3:

' 4.2.4 The proofing after application on the base fabric shall be adequately vulcanized so as to conform to the requirements of this specification.'

( CETDC )

**AMENDMENT NO. 2 APRIL 2005**  
**TO**  
**IS 5915 : 1970 SPECIFICATION FOR SINGLE**  
**TEXTURE RUBBERIZED WATERPROOF FABRICS**

[ Page 7, Table 1, Sl No. (iii) ] — Substitute the following for the existing:

Sl No.	Characteristic	Type 1				Type 2				Method of Test, Ref to
		Quality 1		Quality 2		Quality 1		Quality 2		
		Grade A	Grade B	Grade A	Grade B	Grade A	Grade B	Grade A	Grade B	
iii)	Breaking strength N/5 cm grip, <i>Mix</i> : a) Warp b) Weft	400 350	400 350	400 350	400 350	400 350	400 350	400 350	400 350	7016 (Part 2) : 1981*

\*Methods of test for coated and treated fabrics : Part 2 Determination of breaking strength and extension at break (*first revision*).

(PCD 13)

Reprography Unit, BIS, New Delhi, India